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SPECIFICATION FOR PARAQUAT DICHLORIDE WATER SOLUBLE CONCENTRATES

(First Revision)

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SPECIFICATION FOR PARAQUAT DICHLORIDE WATER SOLUBLE CONCENTRATES

(First Revision)

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(*Continued on page* 10)

AMENDMENT NO. 1 MAY 1994
TO
IS 8497 : 1982 SPECIFICATION FOR PARAQUAT DICHLORIDE WATER SOLUBLE CONCENTRATES

(*Page* 5, *clause* 4.1) — Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause 2.3.1 of the standard.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Indian Standard
SPECIFICATION FOR PARAQUAT DICHLORIDE WATER SOLUBLE CONCENTRATES
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 30 September 1982, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Paraquat dichloride water soluble concentrates are largely used in the control of weeds in agricultural crops.

0.3 Paraquat dichloride water soluble concentrates are generally formulated to contain 17·4 percent (*m/m*) of paraquat cation.

0.4 This standard was first published in 1977. In this revision, besides modifications in the title and testing procedure, opportunity has been taken to express its strength in terms of paraquat cation and to refer its packaging requirements to IS:8190 (Part II)-1980*.

0.5 In the preparation of this standard due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these rules, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

*Requirements for packing of pesticides: Part II Liquid pesticides (*first revision*).

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for paraquat dichloride water soluble concentrates.

2. REQUIREMENTS

2.1 Description — The material shall essentially be an aqueous solution of paraquat dichloride, with or without added surface active agents and corrosion inhibitors, and shall be free from immiscible solvents, sediment and suspended matter.

2.2 Paraquat dichloride used for formulation of water soluble concentrate shall be of 42 percent (*m/m*) minimum purity in terms of paraquat dichloride salt.

2.3 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR PARAQUAT DICHLORIDE WATER SOLUBLE CONCENTRATES

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO APPENDIX
(1)	(2)	(3)	(4)
i)	Paraquat cation content, percent by mass	Nominal value as declared on the container (*see* **2.3.1**)	A
ii)	Free 4, 4′ -bipyridyl content percent by mass, *Max*	0·25 percent of the paraquat cation content	B
iii)	Hydrogen ion concentration (*p*H)	6·5 to 7·5	C

2.3.1 When determined by the method prescribed in Appendix A, the observed paraquat cation content, percent by mass of any of the samples shall not differ from the declared nominal value by more than the tolerance limits indicated below:

Nominal Value, Percent	*Tolerance, Percent*	
Up to 9	+ 10 − 5	of the nominal value
Above 9 and below 50	± 5	
50 and above	+ 5 − 3	

2.3.1.1 The actual value of the active ingredient shall be calculated to the third decimal place and then rounded off to the second decimal place before applying the tolerance as stipulated in **2.3.1**

2.3.1.2 The average content of all samples shall not be lower than the nominal value

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as per requirements given in IS.8190 (Part II)-1980*.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to any other information as required under the *Insecticides* Act and Rules:

a) Name of the material,
b) Name of the manufacturer,
c) Batch number,
d) Date of manufacture,
e) Net volume of contents,
f) Nominal paraquat cation content percent (*m/m*), and
g) Minimum cautionary notice worded as in the Insecticides Act and Rules

3.2.1 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard Methods for Sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the matter shall be as agreed to between the concerned parties.

---

*Requirements for packing of pesticides: Part II Liquid pesticides (*first* revision)

5. TESTS

5.1 Tests shall be carried out by appropriate methods as specified in 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS: 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF PARAQUAT CATION CONTENT

A-0. OUTLINE OF METHOD

A-0.1 The sample is treated with alkaline sodium dithionite solution and the intensity of the blue colour developed is measured differentially at 600 nm. The paraquat cation content is obtained by reference to a calibration graph prepared concurrently with series of analysis.

A-1. APPARATUS

A-1.1 Spectrophotometer — With 1 cm glass cells and permanent blue glass reference standard.

NOTE — A neutral glass filter or metal gauges giving an extinction of about 1·5 at 600 nm may be substituted for blue glass reference standard.

A-2. REAGENTS

A-2.1 Sodium Dithionite Solution — One percent (*m/m*) in freshly prepared 0·1 N sodium hydroxide solution.

NOTE — Do not keep the sodium dithionite solution for more than 1½ hours. Sodium dithionite is unstable in presence of moisture. Store in 25 ml bottles with air-tight closures and keep these in a vacuum desiccator containing an efficient dessicant.

---

*Specification for water for general laboratory use (*second revision*).

A-2.2 Paraquat Standard Solution — Weigh 0·1728 g of pure paraquat dichloride previously dried to constant mass at 110°C for five hours, and cool in a desiccator before use. Dissolve in water and dilute to 500 ml with water and make up the volume to 500 ml in a volumetric flask. Prepare the solution fresh when required. One ml of this standard solution contains 0·25 mg of paraquat cation since paraquat dichloride contain 72·3 percent paraquat cation content.

A-3. PROCEDURE

A-3.1 Weigh accurately portion of well-mixed sample containing about 0·8 to 0·9 g of paraquat dichloride, transfer to a 250-ml volumetric flask, dilute to the mark with water and mix. Designate this as solution *A*.

A-3.2 Pipette 10 ml of Solution *A* into a 250 ml volumetric flask, dilute to the mark with water and mix. Designate this as Solution *B*.

A-3.3 Measure 10 ml of Solution *B*, add 6·0, 8·0, 10·0, 12·0 and 14·0 ml portions of standard paraquat solution containing 1·5, 2·0, 2·5, 3·0 and 3·5 mg of paraquat cation into 100-ml volumetric flasks, and dilute the contents of each flask with water to about 80 ml.

A-3.4 Add 10 ml of sodium dithionite solution to the contents of one of the flasks and make up to the mark with water. Mix the contents by inverting the flask end-over-end three times at such a speed that the air bubble travels from one end to the other. Do not shake the flask vigorously as this tends to cause fading of the colour due to the oxidation. Immediately transfer the solution to a 1 cm cell and measure the extinction of the solution at 600 nm relative to the permanent reference standard. Similarly treat each flask in turn, completing the extinction measurement without delay, before adding the dithionite to the next solution. Draw the graph relating differential extinctions of the standard solutions to their paraquat cation content.

A-4. CALCULATION

A-4.1 Paraquat cation content, percent by mass

$$= \frac{250 \times 250 \times 100 \times M_1}{10 \times 10 \times 1000 \times M} = 62{\cdot}5 \times \frac{M_1}{M}$$

where

M_1 = mass, in mg of the paraquat cation content of the sample solution as read from graph; and

M = mass, in g of the sample taken for the test.

APPENDIX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF FREE 4, 4'-BIPYRIDYL CONTENT

B-0. OUTLINE OF THE METHOD

B-0.1 A portion of the sample is made alkaline and extracted with chloroform The extract is reduced to small bulk and then subjected to GLC analysis using 5, 6-benzoquinoline as an internal standard.

B-1. APPARATUS

B-1.1 Gas Chromatograph — The gas chromatograph is operated under the following suggested parameters.

NOTE — Gas flow rate, amplification, temperature or any other parameters may be suitably modified as per the sensitivity requirement of the individual gas chromatograph used.

B-1.1.1 *Type* — dual flame ionisation or equivalent.

B-1.1.2 *Fuel Gas* — hydrogen, flow rate 60 ml/minute.

B-1.1.3 *Carrier Gas* — nitrogen, flow rate 60 ml/minutes.

B-1.1.4 *Excess Fuel Gas* — air, flow rate 600 ml/minute.

B-1.1.5 *Temperature* — 175°C isothermal.

B-1.1.6 *Amplifier-Range* — 10^4.

B-1.1.7 *Columns* — 305 mm glass, 6 25 mm nominal diameter.

B-1.1.8 *Recorder* — 1 m*V* full scale deflection.

B-1.1.9 *Phase* — 5 percent (*m/m*) sodium dodecylbenzene sulphonate and 10 percent (*m/m*) potassium hydroxide on acid-washed 180 to 212 micron celite.

B-2. REAGENTS

B-2.1 5, 6-Benzoquinoline

B-2.2 4, 4'-Bipyridyl

B-2.3 Sodium Hydroxide — 1 N

B-2.4 Chloroform

B-3. PROCEDURE

B-3.1 Transfer 10·0 g sample accurately weighed into a 500-ml separating funnel and add 2 to 3 ml of *N* sodium hydroxide solution. Extract the mixture with 100 ml chloroform, repeating the extraction three times. Combine the chloroform extracts and reduce to small bulk using a water-bath.

B-3.2 Add 9·5 ml of a one percent (*m*/*v*) solution of 5, 6-benzoquinoline and mix well.

B-3.3 Inject 1·0 μl of this solution into the gas chromatograph and use suitable attenuations for achieving reasonable peak height.

B-3.4 Inject 1·0 μl of 4, 4′-bipyridyl standard solution, again at suitable attenuation.

B-3.5 Evaluate the percent peak height ratios for the two solutions based on the same attenuation.

B-4. CALCULATION

B-4.1

$$\text{4, 4'-Bipyridyl content, percent by mass} = \frac{\text{peak height ratio (sample)} \times \text{mg standard}}{\text{peak height ratio (standard)} \times 100}$$

APPENDIX C

[*Table* 1, *Item* (iii)]

DETERMINATION OF HYDROGEN ION CONCENTRATION (*p*H)

C-1. APPARATUS

C-1.1 ***p*H Meter** — standardized against buffer of *p*H 7·0.

C-2. REAGENTS

C-2.1 **Water** — redistilled from all glass apparatus, and boiled to expel all carbon-dioxide just before use.

C-2.2 **Phosphate Buffer Solution** — 0·025M. Dissolve 3·402 g of postassium hydrogen phosphate and 3·549 g of sodim hydrogen phosphate in water.

C-3. PROCEDURE

C-3.1 Take 25 ml of the sample in a small beaker. Determine the *p*H of this solution using the *p*H meter.

(*Continued from page* 2)

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